IS 7539 (1975): Carbaryl, Technical [FAD 1: Pesticides and Pesticides Residue Analysis]

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IS : 7539 - 1975

(Reaffirmed - 2012)

Indian Standard

SPECIFICATION FOR CARBARYL, TECHNICAL

(Second Reprint MARCH 1988)

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Gr 3 *June* 1975

Indian Standard

SPECIFICATION FOR CARBARYL, TECHNICAL

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AMENDMENT NO. 1 SEPTEMBER 1982

TO

IS:7539-1975 SPECIFICATION FOR CARBARYL, TECHNICAL

Alteration

(Page 4, Table I):

a) *Sl No.*(iii) - Substitute the following for the existing matter under respective columns:

(1)	(2)	(3)	(4)	(5)
iii)	Total volatiles, percent by mass, *Max*	1.0	c	-

b) *Sl* No. (iv) - Delete and renumber the subsequent items accordingly.

Reprography Unit, BIS, New Delhi, India

AMENDMENT NO. 2 SEPTEMBER 1991

TO

IS 7539 : 1975 SPECIFICATION FOR CARBARYL, TECHNICAL

(*Page 4, Table* 1) — Substitute the following for the existing table:

TABLE 1 REQUIKEMENTS FOR CARBARYL, TECHNICAL

Sl No.	CHARACTERISTIC	REQUIREMENT	METHOD OF TEST, REF TO	
			Appendix of This Standard	Cl No. of IS 6940 : 1982*
(1)	(2)	(3)	(4)	(5)
i)	Carbaryl content, percent by mass, *Min*	97'0	A	—
ii)	1-naphthol, percent by mass, Max	0·4	B	—
iii)	2-naphthyl methylcarbamate, percent by mass, *Max*	0'05	C	
iv)	Total volatiles, percent by mass. *Max*	1'0	D	—
v)	Acidity (as H_2SO_4), percent by mass, Max	0·05	—	11'3
	OR			
vi)	Alkalinity (as NaOH)	0'05	—	11'3

*Methods of test for pesticides and their formulations (*first revision*).

(*Page 4, clause* 3.1) — Substitute the following for the existing clause:

"3.1 Packing — The material shall be packed in clean and dry containers as prescribed under Sl No. (xiv) of Table 1 in IS 8190 (Part 1): 1988 'Requirements for packing of pesticides: Part 1 Solid pesticides (*second revision*)'."

(*Page 5, clause* 4.1) — Substitute the following for the existing clause:

"Representative samples of the material shall be drawn as prescribed in IS 10946 : 1984 'Methods of sampling of technical grade pesticides'."

(*Page* 5, clause 5.2) — Substitute '(see IS 1070 :1977*)' *for* '(see IS: 1070 -1960*)':

(*Page* 5, *foot-note*) — Substitue 'Specification for water for general laboratory use (*second revision*)' *for* 'Specification for water, distilled quality (*revised*)'.

(Page 11) — Insert the following as 'Appendix C' and renumber the existing 'Appendix C' as 'Appendix D':

'APPENDIX C!

[*Table* '1,. *Item (* iii)]

DETERMINATION OF 2-NAPHTHYL METIIYLCARBAMATE CONTENT

C-l PRINCIPLE

A high performance liquid **chromatograph** having a column packed with silica and with an ultra-violet detector used for the determination of 2-naphthyl methylcarbamate content. **The·** content is determined by comparing the response of the sample solution with that of a **2-naphthyl** methylcarbamate reference standard of known purity.

C-2 APPARATUS

C-2.1 High Performance Liquid Chromatograph (HPLC)

A suitable HPLC instrument with stainless steel column having 25 cm length and 4·6 mm internal diameter, packed with silica (5 μ particle size) shall be used. It shall be fitted with an ultra-violet (UV) detector for measuring the absorption at 223 nm. The other operative conditions shall be:

Mobile phase	— **Hexane:** Isopropyl alcohol (98 : 2, v/v)		
Volume of Injection	— 20 μl		
Flow rate	— 1·5 ml/min, which may be increased, if necessary, when Alpha-carbaryl starts eluting, for faster elution		
Retention time	— **4-chlorophenol**	—	17'0 min
	— Beta-carbaryl	—	48'4 min
	— alpha-carbaryl	—	54'4 min

C-3 REAGENTS

c-3.1 **2-naphthyl** Methylcarbamate Reference Standard — of known purity.

C-3.2 **4-chlorophenol** (Internal Standard) — AR grade.

c-3.3 Dichloromethane — Spectroscopic grade.

c-3.4 Hexane — Spectroscopic grade.

c-3.5 Isopropyl Alcohol — Spectroscopic grade.

C-3.6 Mobile Phase — Mix hexane and isopropyl alcohol in the ratio of98 : 2 (v/v).

C-4 PROCEDURE

C-4.1 Preparation of Internal Standard Solution

Weigh accurately about 65-70 mg 4-chlorophenol into a 100-ml Volumetric flask and add 5 ml dichloromethane to dissolve and make up the volume with mobile phase.

C-4.2 Preparation of 2-naphthyl Methylcarbamate Reference Standard Solution

Weigh accurately about 20 mg of the reference standard into a 100-ml volumetric flask, add 5 ml dichloromethane to dissolve and make up the volume with mobile phase. Take 5 ml of this solution into another 100-ml volumetric flask with the help of a pipette and make up the volume with mobile phase. Take 5 ml of this solution into another 100-ml volumetric flask, add 2 ml internal standard solution and make up the volume with mobile phase.

C-4.3 Preparation of Sample Solution

Weigh accurately about 500 mg carbaryl, technical sample into a 100-ml volumetric flask, add 20 ml dichloromethane to dissolve. Add 2 ml internal standard solution and make up the volume with mobile phase.

C-4.4 Estimation

Inject 20 μl of reference standard solution into the HPLC unit and from the integrator, print out and note down the peak heights/areas of the reference standard and internal standard. Adjust the attenuation in such a way that peaks are obtained within the scale in both cases. Inject 20 μl of sample solution and obtain the peak height/area. Compute the percentage of 2-naphthyl methylcarbamate content in the sample by the following formula.

C-5 CALCULATION

$$\text{2-naphthyl methylcarbamate, content, per cent by mass} = \frac{m_1 \times A_2 \times A_3}{A_1 \times m_2 \times A_4} \times P$$

where

m_1 = mass of 2-naphthyl methylcarbamate in reference standard solution;

A_2 = area of 2-naphthyl methylcarbamate peak in sample solution;

A_3 = area of internal standard peak in reference standard solution;

P = percent purity of 2-naphthyl methylcarbamate reference standard;

A_1 = area of 2-naphthyl methylcarbamate peak in reference standard solution;

m_2 = mass of carbaryl, technical sample taken for the test; and

m_4 = area of internal standard peak in sample solution.'

(FACII 0)

Printed at Printrade, New Delhi, India

AMENDMENT NO. 3 MAY 1994
TO
IS 7539 : 1975 **SPECIFICATION** FOR CARBARYL, TECHNICAL

(*Page 5, clause* **4.1**) — Substitute the following for the existing:

'When freshly manufactured material in bulk quantity is offered for inspection, representative samples of the material shall be drawn and tested as prescribed in IS 10627 : 1983 within 90 days of its manufacture. When the material is offered for inspection after 90 days of its manufacture, sampling shall be done as prescribed in IS 10627 : 1983. However, the criteria for conformity of the material when tested, shall be the limits of tolerances, as applicable over the declared nominal value and given under clause 2.2 of the standard.'

(*Page* 6, *clause* A-1.2.1, ■■ ■)— Incorporate the following after the line 10:

'In order to avoid overheating, a thermostatically controlled heating mental shall be used '

(FAD 1)

Reprography Unit, BIS, New Delhi, India

AMENDMENT NO. 4 MAY 1996
TO
IS 7539 : 1975 SPECIFICATION FOR CARBARYL, TECHNICAL

[*Page 5, clause 4.1 (see also Amendment No. 3)*] — **Delete the text 'when freshly manufactured ..... of the standard'.**

[*Page 5, clause* 5.2 (see *also Amendment No. 2)*] — **Substitute '(see IS 1070 :1992*)' *for* '(see IS : 1070 - 1977*)'.**

(*Page* 5, *foot-note marked* '*') — **Substitute** 'Reagent grade water (*third revision*)' **for the existing title.**

(FAD 1)

Reprography Unit, BIS, New Delhi, Ida

AMENDMENT NO. 5 OCTOBER 2010
TO
IS 7539 : 1975 SPECIFICATION FOR CARBARYL, TECHNICAL

[Page 5, *clause* 4.1 (*see also Amendments No.* 3 *and* 4)] — Substitute the following for the existing:

'4.1 Representative samples of the material shall be drawn as prescribed in IS 10946:1996**'.

(.Page 5, footnote) — Add the following after the existing footnote:

'••Methods of sampling for technical grade pesticides *(first revision).'*

(FAD 1)

Reprography Unit, BIS, New Delhi, India

Indian Standard

SPECIFICATION FOR CARBARYL, TECHNICAL

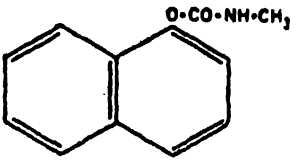
0. FOREWORD

OS This Indian Standard was adopted by the Indian Standards Institution on 2% January 1975, after the draft finalized by the Pest Control Sectional Committee had been approved by the Agricultural and Food Products Division Council and the Chemical Division Council.

0.2 Carbaryl, a carbamate insecticide, is used in the preparation of formulations required for the control of many pests, and is specially useful in the control of pests of cotton, fruits and vegetables.

0.2.1 Carbaryl is the accepted common name by the International Organization for Standardization for the insecticidal chemical containing essentially **l-naphthyl** methylcarbamate. The empirical and structural formulae and molecular weight of this product are given below:

Empirical *Formula*	*Structural Formula*	*Molecular Weight*
$C_{12}H_{11}NO_2$		201·2



0.3 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with ES : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

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1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for carbaryl, technical employed in the preparation of insecticidal formulations.

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*Rules for rounding off numerical values (*revised*).

2. REQUIREMENTS

2.1 Description — The material shall be in the form of crystalline solid, slightly coloured ranging from light grey to white.

2.2 The material shall comply with the requirements as specified in Table 1.

TABLE 1 REQUIREMENTS FOR CARBARYL, TECHNICAL

Sl No.	Characteristic	Requirement	Method of Test, Ref to	
			Appendix	Cl No. of IS : 6940-1973*
(1)	(2)	(3)	(4)	(5)
i)	Carbaryl content, percent by mass, *Min*	97	A	
ii)	1-Naphthol, percent by mass, Max	0·4	B	—
iii)	Volatiles, percent by mass, *Max*	0·5	C	—
iv)	Water, percent by mass, *Max*	0·1	—	4.1
v)	Acidity (as H_2SO_4), percent by mass, *Max*	0.05	—	11.3
	or			
vi)	Alkalinity (as NaOH), percent by mass, Max	0·05	—	11.3

*Methods of tests for pesticides and their formulations.

3. PACKING AND MARKING

3.1 Packing — Carbaryl, technical shall be packed in clean and dry multiwall polycoated paper bags (6 ply) or in hessian bags which may be either bitumenized, or lined with kraftpaper or suitable low density polyethylene film.

3.2 **Marking** — The containers shall be securely closed and shall bear legibly and indelibly the following information in addition to the provisions under the Insecticides Act and Rules:

a) Name of the material;
b) Name of the manufacturer;
c) Date of manufacture;
d) Batch Number;
e) Net mass of contents;
f) Active ingredient (**Carbaryl**) content, percent by mass; and

g) The minimum cautionary notice worded as under:

'HARMFUL IF SWALLOWED. AVOID EXCESSIVE INHALATION OR SKIN CONTACT. AVOID CONTAMINATION OF FOODSTUFFS, EMPTY FOODSTUFF CONTAINERS AND ANIMAL FEEDS. KEEP OUT OF REACH OF CHILDREN. IF POISONING OCCURS, CALL A PHYSICIAN. ATROPINE AND OXYGEN ARE USEFUL IN TREATMENT. '

3.2.1 The containers may also be marked with the Standard Mark.

NOTE – The use of the Standard Mark is governed by the provisions of the Bureau of Indian Standards Act, 1986 and the Rules and Regulations made thereunder. The Standard Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well defined system of inspection, testing and quality control which is devised and supervised by BIS and operated by the producer. Standard marked products are also continuously checked by BIS for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

4. SAMPLING

4.1 Representative samples of the material for ascertaining conformity to the requirements of this specification shall be drawn as prescribed in ' Indian Standard methods of sampling of pesticides and their formulations ' (*under preparation*).

NOTE — Until the standard under preparation is published, the matter shall be as agreed to between the concerned parties.

5. TESTS

5.1 Tests shall be carried out as prescribed in the appropriate appendices and clauses, as specified in col 4 and 5 of Table 1.

5.2 **Quality of Reagents** — Unless specified otherwise, pure chemicals and distilled water (see IS : 1070-1960*) shall be employed in tests.

NOTE — ' Pure chemicals ' shall mean chemicals that do not contain impurities which affect the results of analysis.

APPENDIX A

[*Table* 1, *Item* (i)]

DETERMINATION OF CARBARYL CONTENT

A-0. GENERAL

A-0.1 For the determination of Carbaryl content two methods namely titration method (see A-1) and colorimetric method (see A-2) have been

*Specification for water, distilled quality (*revised*).

specified. Either one of these methods may be used for routine testing but the colorimetric method shall be used as a referee method in case of dispute.

A-1. TITRATION METHOD

A-1.1 Reagents

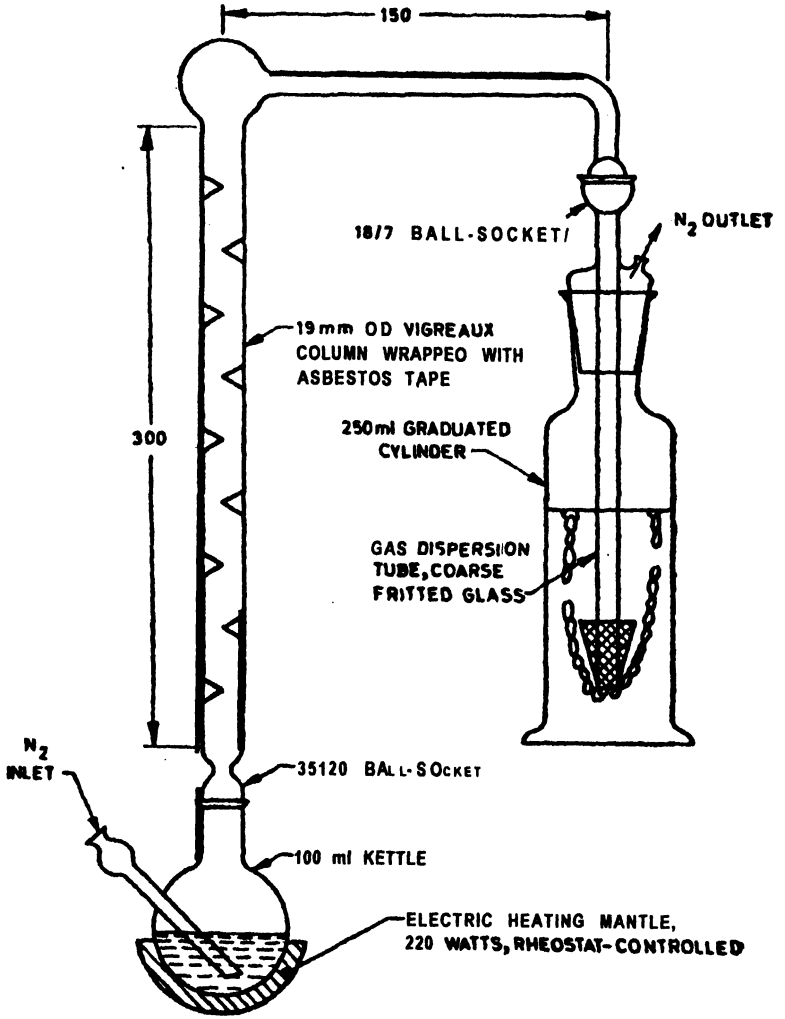
A-1.1.1 *Standard Potassium Hydroxide Solution* — 1·0 *N*, prepared by dissolving 66 g of potassium hydroxide in 40 ml of distilled water. Dilute to one litre with diethylene glycol.

A-1.1.2 *Dilute Boric Acid Solution* — 2 percent. Dissolve 20 g of boric acid in distilled water and dilute to one litre. Heat to 70°C, swirl, and cool to room temperature. Add 10 ml of 0·1 percent bromocresol green indicator and neutralize to a green end point with O-1 N hydrochloric acid.

A-1.1.3 *Standard Hydrochloric Acid Solution* — 0·1 N, aqueous.

A-1.2 Procedure

A-1.2.1 Assemble the apparatus as shown in Fig. 1. Introduce 0·4 to 0·5 g of the sample accurately weighed to the nearest 0·1 mg into the 100-ml distillation flask. By means of a graduated cylinder, add 50 ml of the standard potassium hydroxide solution to the flask and add a few glass beads to ensure smooth ebulition. Apply silicone grease to the ball joint



All dimensions in millimetres.

FIG. 1 APPARATUS FOR DETERMINATION OF CARBARYL CONTENT (TITRATION METHOD)

A-1.3 Calculation

A-1.3.1 Carbaryl content, percent by mass = $20{\cdot}12 \times \frac{A \times N}{M}$

where

A = volume in ml of standard hydrochloric acid solution required for the titration of the sample,

N = normality of the standard hydrochloric acid solution, and

M = mass in g of the sample taken for the test.

A-2. COLORIMETRIC METHOD

A-2.1 Principle — The material is heated with methonolic sodium hydroxide. To the resultant 1-naphthol, iodine solution (see A-2.2.3) is added and the violet colour thus produced is measured in a colorimeter and the carbaryl content calculated by running a sample of analytical grade carbaryl alongwith the material under analysis.

A-2.2 Reagents

A-2.2.1 *Methyl Alcohol* — distilled, boiling range, 64 to 65°C.

A-2.2.2 *Methanolic Sodium Hydroxide Solution* — 2 percent. Prepared by weighing 10 g of sodium hydroxide pellets, dissolving in 10 ml of water and making up 500 ml with distilled methanol in a volumetric flask.

A-2.2.3 *Colour Reagent Solution* — prepared by dissolving 1·27 g of iodine, 4 g of potassium iodide in 50 ml water in a beaker. This is then transferred to a 100-ml volumetric flask, shaken well and made to mark with water. It is to be ensured that all iodine goes into solution. This solution is diluted ten times its volume in water. This diluted solution is used for developing colour.

A-2.3 Procedure

A-2.3.1 *Preparation of Sample Solution* — Weigh enough material containing carbaryl close to 0·06 g in a 100-ml Erlenmeyer flask. Pipette exactly 1 ml of standard methanolic sodium hydroxide solution followed by 10 ml methyl alcohol into the flask. Heat to boil. Transfer the solution quantitatively into 100-ml volumetric flask. Wash the Erlenmeyer flask with methyl alcohol two or three times and add washings to the volumetric flask and finally make up to the mark with methyl alcohol. Pipette out 5 ml of the above stock solution into 500-ml volumetric flask. Add 300 ml of water and 20 ml of colour reagent solution, and finally make up to mark with water. Shake well.

A-2.3.2 *Preparation of Standard Carbaryl Solution* — Weigh carbaryl, analytical grade (see Note) close to 0·06 g in 100-ml Erlenmeyer flask and proceed as described in A-2.3.1.

NOTE — Carbaryl, analytical grade, may be prepared by repeated recrystallization, three times of carbaryl, technical, from toluene.

A-2.3.3 *Preparation of Blank Solution*- Weigh the same amount of carbaryl, technical that is equivalent to the sample weighed for analysis of carbaryl content. Transfer to 100-ml volumetric flask and make up to the mark with methyl alcohol. Shake well. Pipette out 5 ml of this solution to 500-ml volumetric flask containing 20 ml of colour reagent solution and 300 ml of water. Make up to the 500-ml mark with water. Shake well and use this solution as blank.

A-2.3.4 *Measurement of Absorbance* — Take the above three solutions to the colorimeter and fill one of the cells with the blank solution and adjust the colorimeter to zero absorbance at wavelength of 540 nm. Fill another cell with standard carbaryl solution and measure the optical density at 540 nm. Similarly, measure the optical density for the sample solution.

NOTE- Measurement of optical density shall be carried within 15 minutes after addition of colour reagent.

A-2.4 Calculation

A-2.4.1 Carbaryl content, percent by mass = $\frac{A \times B}{C \times D} \times 100$

where

A = mass in g of the material in standard solution,

B = optical density of sample,

C = mass in g of the material in sample solution, and

D = optical density of the standard solution.

APPENDIX B

[*Table, Item* (ii)]

DETERMINATION OF I-NAPHTHOL

B-1. PRINCIPLE

B-1.1 This method is based on the reaction of I-Naphthol with p-nitrobenzene-diazonium fluoborate (*see* R-1.2.1) to form a coloured compound with an absorbance maximum at 475 nm. The intensity of the colour produced is measured on a spectrophotometer.

E1.2 Reagents

B-1.2.1 *Colour Reagent Solution* — prepared by dissolving 0·025 g of p-nitrobenzene diazonium fluoborate in 25 ml of a solution composed of one part of glacial acetic acid and one part of redistilled dimethyl formamide. Prepare this solution immediately before use.

B-1.2.2 *1-Naphthol* — (Analytical Grade M.P. 96°C.)

B-1.2.3 *Acetone* — redistilled.

B-1.3 Procedure

B-1.3.1 Introduce 0·08 to 0·12 g of the sample weighed to the nearest 0·1 mg into a 100-ml glass-stoppered volumetric flask and dilute to the mark with acetone. Mix thoroughly. Pipette 5 ml of the dilution into each of four 250-ml glass-stoppered Erlenmeyer flasks. Pipette 15 ml of glacial acetic acid into each flask and mix thoroughly. Reserve two of the flasks for the blank determination. Into each of the other flasks pipette 1 ml of the colour reagent solution while swirling the flasks. Allow the flasks to stand at 25 ± 3°C for exactly 6 minutes. Transfer a portion of the blank and sample solutions to respective 1·0 cm cells of the spectrophotometer and obtain the absorbance of the sample at a wavelength of 475 nm based on a reading of zero for the blank. From a previously prepared calibration curve read the total mg of 1-Naphthol corresponding to the absorbance.

B-1.3.2 *Calibration Curve* — Prepare a standard solution by introducing 0·1 g of l-Naphthol, weighed to the nearest 0·1 mg into a 1 000-ml glass-stoppered volumetric flask, dilute to the mark with glacial acetic acid and mix thoroughly. Pipette 10 ml of the solution into a 50-ml glass-stoppered volumetric flask, dilute to the mark with glacial acetic acid, and mix thoroughly. One ml of this solution contains 0·02 mg of I-Naphthol.

Pipette 0·5, 1·0, 2·0 and 3·0 ml aliquots of the second dilution into respective 250-ml Erlenmeyer flasks. Reserve a fifth flask as a blank. From a burette, add sufficient acetic acid to make a total volume of 20 ml. Into each flask, pipette one ml of the colour reagent solution while swirling the flask. Obtain the absorbance of each standard following the procedure mentioned earlier for the sample (see B-1.3.1). Plot a curve of absorbance versus 1-Naphthol concentration in mg. The absorbance for the 0·04 mg standard should be opproximately 0·53 and the curve should intersect the origin.

B-2. CALCULATION

B-2.1 I-Naphthol, percent by mass $= \frac{A \times 2}{M}$

where

A = 1-Naphthol, mg, from calibration curve; and
M = mass in g of sample taken for the test.

APPENDIX C

[*Table* 1, Item (iii)]

DETERMINATION OF VOLATILES

C1. PROCEDURE

C-1.1 Weigh 10 g of material to the nearest 0·1 mg in a tared weighing dish. Place the dish in an oven maintained at 105-1 10°C for one hour. Remove the dish from the oven, allow to cool in a desiccator and reweigh to the nearest 0·1 mg.

C-2. CALCULATION

C-2.1 Volatiles, percent by mass = $100 - \frac{m}{M} \times 100$

where

m = mass in g of residue after drying, and

M = mass in g of material taken for the test.

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